

GOLUBTSOV, S. A.

I. V. Trofimova, K. A. Andrianov and S. A. Golubtsov, "The Synthesis of Trichlorsilane."

Report presented at the Second All-Union Conference on the Chemistry and Practical Application of Silicon-Organic Compounds held in Leningrad from 25-27 September 1958.

Zhurnal prikladnoy khimii, 1958, Nr 1, pp 238-240 (USSR)

ANDRIANOV, K.A.; GOLUBTSOV, S.A.; TISHINA, N.N.; TROFIMOVA, I.V.

Direct synthesis of phenyltrichlorosilane in a fluidized bed.  
Zhur.prikl.khim. 32 no.1:201-207 Ja '59. (MIRA 12:4)  
(Silane)

5.3600

75687  
SOV/80-32-10-36/51

AUTHORS: Andrianov, K. A., Golubtsov, S. A., Trofimova, I. V.,  
Lobusevich, N. P.

TITLE: Direct Synthesis of Methylchlorosilanes in a Fluidized  
Bed

PERIODICAL: Zhurnal prikladnoy khimii, 1959, Vol 32, Nr 10, pp  
2332-2335 (USSR)

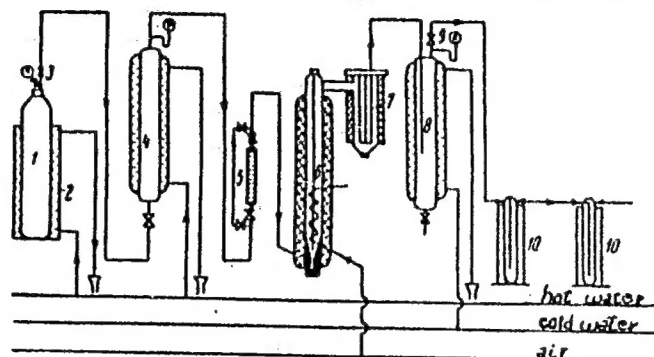
ABSTRACT: The present work was done in 1954-1955. The effective-  
ness of the fluidized bed application was checked by  
the synthesis of methylchlorosilanes. The reaction  
between methyl chloride and silicon was carried out in the  
presence of a silicon-copper alloy (20% Cu), at 4-5  
atmospheres pressure. The reaction is exothermic and  
needs to be cooled. Special apparatus was constructed  
which included a cooling system. Dimethyldichlorosilane  
content was between 42 and 47% in the reaction mix-  
ture. A schematic diagram of the apparatus is given,  
where 1 is methyl chloride cylinder; 2 is water bath;

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3 is valve; 4 is evaporator, heated with hot water; 5 is rotameter, 6 is reactor, 7 is filter; 8 is water-cooled trap; 9 is valve; 10 is traps cooled with dry ice and acetone. There are 2 figures; 2 tables; and 4 Soviet references.  
May 15, 1958

SUBMITTED:  
Card 2/2

5.3700(B)

5(3)

AUTHORS:

Golubtsov, S. A.,

Andrianov, K. A., Corresponding Member, AS USSR,

Tishina, N. N.

68812  
S/020/60/131/01/025/060  
B011/B006

TITLE:

Reaction of Joint Phenylation of Trichlorosilane and Silicon  
Tetrachloride

PERIODICAL:

Doklady Akademii nauk SSSR, 1960, Vol 131, Nr 1, pp 91-93  
(USSR)

ABSTRACT:

The authors intended to eliminate the side reactions which lower the yield to 40% theoretical phenyltrichlorosilane (Ref 5), and at the same time tried to phenylate the silicon tetrachloride formed in the reaction. They found that the hydrogenation of silicon tetrachloride with hydrogen proceeds satisfactorily, if the reagents are heated under the same conditions as bring about the phenylation of trichlorosilane (440-460°, 180 atm). The results obtained proved that it is fundamentally possible to phenylate  $\text{SiCl}_4$ , if it is first hydrogenated to trichlorosilane, and only then reacted with benzene. The hitherto unused hydrogen formed as a by-product

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Reaction of Joint Phenylation of Trichlorosilane  
and Silicon Tetrachloride

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B011/B006

in reaction (1) was utilized for the first stage of this process. For this purpose, the authors reacted a mixture of  $\text{SiCl}_4$ ,  $\text{C}_6\text{H}_6$ , and trichlorosilane (Ref 6) under the above-mentioned reaction conditions. The molar ratio of trichlorosilane :  $\text{SiCl}_4$  was varied between 0.25 : 0.75 and 0.85 : 0.15.

It can be seen from figure 1 that the yield in phenyltrichlorosilane (in g-mol per 100 g-mol of reacted trichlorosilane) increases with increasing content of  $\text{SiCl}_4$  in the reaction

mixture. This cannot be explained by the suppression of the disproportionation of trichlorosilane, occurring as a side reaction, since the yield in phenyltrichlorosilane often considerably exceeds 100 g-mol per 100 g-mol trichlorosilane. This proves that the phenylation proceeds according to the intended scheme (see scheme given), under utilisation of the hydrogen formed in reaction (1). The increased hydrogen pressure facilitates the first reaction, i.e. hydrogenation. For this reason phenyltrichlorosilane, final product, was obtained in much higher yield than is obtainable at at-

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Reaction of Joint Phenylation of Trichlorosilane  
and Silicon Tetrachloride

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atmospheric pressure. The authors have thus proved that the phenylation of  $\text{SiCl}_4$  with benzene gives sufficiently high yields even without use of metalorganic compounds, if conditions are so chosen, that  $\text{SiCl}_4$  is hydrogenated by hydrogen to the intermediate trichlorosilane according to reaction (2). Trichlorosilane then reacts with benzene and forms phenyltrichlorosilane, regenerating hydrogen. There are 1 figure and 6 references, 5 of which are Soviet.

SUBMITTED: November 5, 1959

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S/661/61/000/006/003/081  
D205/D302

AUTHOR: Golubtsov, S. A.

TITLE: Direct synthesis of organic chlorosilanes

SOURCE: Khimiya i prakticheskoye primeneniye kremneorganicheskikh soedineniy; trudy konferentsii. no. 6, Doklady, diskussii, resheniye II Vses. konfer. po khimii i prakt. prim. kremneorg. soyed., Len., 1958. Leningrad. Izdvo AN SSSR, 1961, 24-25

TEXT: The need for a more economical design of a process for direct synthesis of chlorosilanes is stressed. Two main problems are to be solved. One is the possibility of regulating the composition of the methyl chlorosilanes. The second is the design of new processes for various alkyl- and aryl chlorosilanes (including products having a functional group in the radical) based on by-products of the direct synthesis or some other readily available raw materials. There is much room for the USSR scientist in the fields of mechanisms and kinetics of the processes and also in the field of preparing new catalysts. ✓

Card 1/1



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5.3700

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S/661/61/000/006/004/081  
D205/D302

AUTHORS: Trofimova, I. V., Andrianov, K. A., Golubtsov, S. A.,  
Turetskaya, R. A., Belyakova, Z. V., Yakusheva, T. M.,  
Lobusevich, N. P. and Luzganova, M. A.

TITLE: On the regulation of the composition of products in the  
direct synthesis of methyl- and ethyl chlorosilanes in  
a fluidized bed

SOURCE: Khimiya i prakticheskoye primeneniye kremneorganiches-  
kikh soyedineniy; trudy konferentsii. no. 6, Doklady,  
diskussii, resheniye. II Vses. konfer. po khimii i prakt.  
prim. kremneorg. soyed., Len., 1958. Leningrad, Izd-vo  
AN SSSR, 1961, 25-27

TEXT: Regulation of the process is one of the main problems in  
preparing monomeric organosilicon compounds. The most interesting  
results were obtained during the attempt to regulate the product  
composition by varying the preparation procedure of the catalyst.

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On the regulation ...

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D205/D302

This method opens wide possibilities as can be judged from the obtained data. Thus a synthesis carried out on a Si-Cu melt containing 15 - 20% Cu gave 6%  $\text{CH}_3\text{HSiCl}_2$ , 30 - 40%  $(\text{CH}_3)_2\text{SiCl}_2$  and 40%  $\text{CH}_3\text{SiCl}_3$ , while the synthesis on a Si-Cu melt activated by cuprous chloride gave 6%  $\text{CH}_3\text{HSiCl}_2$ , 55%  $(\text{CH}_3)_2\text{SiCl}_2$  and 25%  $\text{CH}_3\text{SiCl}_3$ . Further modifications of the catalyst bring about further changes in the composition. Preliminary experiments on the production of methyl chlorosilanes from methane, were performed. Methyl dichlorosilane can be prepared in this way, with trichlorosilane and silicon by-products which can be utilized. For synthesis of ethyl chlorosilanes other methods of regulating the product composition were employed: Preliminary treatment of the Si-Cu catalyst by various gases at elevated temperatures, dilution of ethyl chloride by gases and activation of the ethyl chloride by minor additions. The most interesting results were obtained with preliminary treatment by air at  $370^\circ\text{C}$ . About 45% of diethyl chlorosilane was present in the product using a catalyst treated in this way. Dilution

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On the regulation ...

S/661/61/000/006/004/081  
D205/D302

of EtCl with HCl and the introduction of 0.5 - 0.7% moisture increases the ethyl dichlorosilane content of diethyl dichlorosilane. There are 1 figure and 3 tables.

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37754

S/661/61/000/006/005/031  
D205/D302

5: 3700  
11. 1250

AUTHORS: Lobusevich, N. P., Trofimova, I. V., Andrianov, K. A.,  
Golubtsov, S. A. and Belyy, A. P.

TITLE: Influence of some metal additives on the activity of  
silicon-copper alloys in the synthesis of methyl chloro-  
silanes

SOURCE: Khimiya i prakticheskoye primeneniye kremneorganiches-  
kikh soyedineniy; trudy konferentsii. no. 6, Doklady,  
diskussii, resheniye. II Vses. konfer. po khimii i  
prakt. prim. kremneorg. soyed., Len., 1958. Leningrad.  
Izd-vo AN SSSR. 1961, 28-31

TEXT: The influence of impurities commonly encountered in silicon  
(Al, Fe, Ca) and copper (Bi, Sn, Pb) on the activity of silicon-  
copper alloys used in methyl chlorosilane synthesis was investiga-  
ted. Two series of alloys were prepared: 1) From purified Si with  
less than 0.2% of impurities; 2) from Kp-1 (Kr-1) silicon with 2%  
impurities. These alloys, notwithstanding the identical procedure

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D205/D302

Influence of some ...

of preparation, were entirely different in their activity. Thus, the alloys prepared from the purified Si gave a much lower dimethyl dichlorosilane yield than those made of the non-purified Si. The average figures were 34.0% and 41.0% respectively. The introduction of Al (up to 1.5%), Fe (up to 3%), Ca (up to 0.6%), each taken separately, had very little influence on the activity of the alloys prepared from purified and non-purified Si. The use of Kr-2 silicon gives worse results. Pb and Bi have a strong detrimental influence on the activity of the alloys even at a concentration of 0.01% only, while the results obtained on the introduction of Sn were irreproducible. There are 9 tables.

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S/661/61/000/006/014/081  
D205/D302

AUTHOR: Golubtsov, S. A.

TITLE: Continuous synthesis of phenyl trichlorosilane

SOURCE: Khimiya i prakticheskoye primeneniye kreneorganicheskikh soyedineniy; trudy konferentsii, no. 6, Doklady, diskussii resheniye. II Vses. Konfer. po khimii i prakt. prim. kremneorg. soyed., Len. 1958. Leningrad. Izd-vo AN SSSR, 1961, 85-86

TEXT: In 1958, an apparatus for the continuous synthesis of phenyl trichlorosilane was put into production. Benzene and trichlorosilane are fed through a filter into a mixer which is connected via a cooler and surge capacitor with a column for the absorption of outlet gases. The mixture goes through filters and a high-pressure pump into a 40 liter reaction column working under high pressure and electrically heated. The products are discharged through a series of valves and a cooler into a receiver. The gases are led into the absorber and the products are collected in a capacity tank and from

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Continuous synthesis of ...

S/661/61/000/006/014/081  
D205/D302

there fed to rectification. Data of 4 runs ranging in duration from 45 to 98 hours are tabulated showing that the laboratory yields are fully reproduced in continuous operation. Indications are that by appropriate changes in the synthesis conditions a quantitative yield of phenyl trichlorosilane can be achieved. There are 1 figure and 1 table.

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S/661/61/000/006/016/081  
D205/D302

AUTHORS: Popeleva, G. S., Trofimova, I. V., Andrianov, K. A.  
and Golubtsov, S. A.

TITLE: Study of vinyl chlorosilane synthesis

SOURCE: Khimiya i prakticheskoye primeneniye kremneorganicheskikh soedineniy; trudy konferentsii, no. 6, Doklady, diskussii resheniye. II Vses. Konfer. po khimii i prakt. prim. kremneorg. soyed., Len. 1958. Leningrad. Izd-vo AN SSSR. 1961, 90-94

TEXT: During the investigation of the reaction  $3\text{CH}_2 = \text{CHCl} + \text{SiCH}_2 = \text{CHSiCl}_3$  it was found that the catalyst prepared from precipitated  $\text{CuO}$ ,  $\text{Si}$  powder and a liquid glass binder was the most active. 3 methods of contacting were tried: (1) Stationary bed of pelletized catalyst (2 - 3 mm pellets); (2) an agitated powder bed; (3) a fluidized bed. The first method gave good results when using anhydrous  $\text{FeCl}_3$  as an activator. The second method allowed the low-  
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Study of vinyl ...

S/661/61/000/006/016/031  
D205/D302

ering of the temperature from 460 to 420°C. The vinyl trichlorosilane yield was 33% and the output 10 - 15 g/hour/kg of catalyst. The third method resulted in a reduction of the contact time by a factor of 12 and a corresponding considerable increase in temperature. The yield of vinyl trichlorosilane was reduced, as the side reactions were enhanced, to ~26%. In contrast to the synthesis of methyl, ethyl and phenyl chlorosilanes where the best results are obtained in the fluidized bed, this method does not provide the contact times necessary for synthesis of vinyl chlorosilanes. As an alternative to the above reaction, the reaction  $\text{HSiCl}_3 + \text{CH}_2 = \text{CHSiCl}_3 + \text{HCl}$  is proposed. This reaction was investigated, yields of 65% being obtained at 500°C with a contact time of 35 seconds. In the synthesis of methyl vinyl dichlorosilane by the reaction  $\text{CH}_3\text{SiHCl}_2 + \text{CH}_2 = \text{CHCl} \rightarrow \text{CH}_3(\text{CH}_2=\text{CH})\text{SiCl}_2 + \text{HCl}$ , the optimum conditions ensuring a 55% yield were 540°C and a contact time of 30 seconds. Thus the condensation of hydrochlorosilanes with vinyl

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Study of vinyl ...

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chloride gives a simple continuous' method for preparing vinyl tri-chlorosilane and methyl vinyl dichlorosilane. There are 3 figures and 2 tables.

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X

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S/661/61/000/006/019/081  
D205/D302

AUTHORS: Tarasova, A. S., Petrov, A. D., Andrianov, K. A., Golubtsov, S. A., Ponomarenko, V. A., Cherkayev, V. G., Zadorozhnyy, N. A. and Vavilov, V. V.

TITLE: Continuous addition of hydrochlorosilanes to unsaturated compounds

SOURCE: Khimiya i prakticheskoye primeneniye kremneorganicheskikh soyedineniye; trudy konferentsii, no. 6, Doklady, diskussii resheniye. II Vses. Konfer. po khimii i prakt. prim. kremneorg. Soyed., Len. 1958. Leningrad, Izd-vo AN SSSR. 1961, 99-100

TEXT: For practical application of the addition reactions of methyl dichlorosilane, ethyl dichlorosilane and trichlorosilane to liquid and gaseous unsaturated compounds an apparatus was designed and optimum conditions of synthesis were established. The chlorosilane and the gas are fed into a reactor. The products are discharged via a cooler into a receiver equipped with a reflux. Dur-

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Continuous addition of ...

S/661/61/000/006/019/081  
D205/D302

ing the reaction the reactor and cooler are cooled by water, the receiver and the reflux by brine. The arrangement was tested on the reaction of ethylene with methyl dichlorosilane and ethyl dichlorosilane. The experiments have shown that in the 35 - 200°C temperature range the reaction is unchanged giving a 65 - 75% yield. No by-products are formed and the output is high (> 6 kg of methyl ethyl dichlorosilane/hr/l of reactor volume). The process is amenable to automation owing to its insensitivity to temperature changes. There are 1 figure and 1 table.

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21421

S/191/61/000/012/005/007

B110/B147

AUTHORS: Golubtsov, S. A., Belyakova, Z. V., Yakusheva, T. M.TITLE: Synthesis of  $\beta$ -ethyl cyanide trichlorosilane

PERIODICAL: Plasticheskiye massy, no. 12, 1961, 20 - 21

TEXT: The long heating in the synthesis of  $\beta$ -ethyl cyanide trichlorosilane (I) according to J. C. Saam, J. L. Speier (see below): $\text{HSiCl}_3 + \text{CH}_2=\text{CH-CN} \longrightarrow \text{Cl}_3\text{SiCH}_2\text{CH}_2\text{CN}$  was avoided by working in a 0.5

liter autoclave. 53 g of acrylonitrile, 181.5 g of trichlorosilane, and 3.6 g of dimethyl formamide were heated for 1 - 2 hr, and the reaction products fractionated. Optimum yield (60 - 67%) of I was obtained by 1-hr heating at 80 - 100°C. At 50 - 70°C 47%, at 110 - 130°C 53% yield, only the  $\beta$ -isomer, was obtained. Rectification on a column (efficiency = 15 theoretical plates) yielded a fraction with 3.2% of trichlorosilane, 2.7% of azeotropic mixture (11% of acrylonitrile and 89% of  $\text{SiCl}_4$ ), and 81.3% of  $\text{SiCl}_4$ . The gases contained 84% of  $\text{H}_2$ , 9.4% of  $\text{N}_2$ , 1.5% of acid

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S/191/61/000/012/005/007  
B110/B147

Synthesis of  $\beta$ -ethyl cyanide....

admixtures, and 5.1% of  $\text{CH}_3\text{Cl}$ . For the continuous synthesis of I (Fig.), dosing vessel 1 was filled through opening 4. The reaction mixture is pressed into the reaction vessel 2 (a spiral pipe immersed into water) by  $\text{N}_2$  supplied through 5. Dosing valve 7 and connector 8 are placed between 1 and 2. The mixture passes from 2 into condenser 3. The condensate reaches the receiving vessel 12. The noncondensed gases are carried off through the throttle valve 9. By means of the continuous apparatus which can easily be automatized, working is possible for a longer period under steady conditions. Productivity of the reaction vessel per unit volume increases by the twofold as compared to cyclic operation under pressure, and by the 140-fold as compared to operation under atmospheric pressure. There are 1 figure and 5 non-Soviet references. The three most recent references to English-language publications read as follows: G. D. Cooper, M. Prober, J. Org. Chem., 25, 240 (1960); J. C. Saam, J. L. Speier, J. Org. Chem., 24, 427 (1959); S. Nozakura, S. Konotsune, Bull. Chem. Soc. Japan, 29, 322 (1956).

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15.8170

27902

S/079/61/031/010/002/010  
D227/D302

AUTHORS: Belyakova, Z.V., and Golubtsov, S.A.

TITLE: Synthesis of some (chloro-organo) silanes

PERIODICAL: Zhurnal obshchey khimii, v. 31, no. 10, 1961,  
3178-3181

TEXT: Tri- substituted silanes in which a silicon atom is linked to both hydrogen and a chlorinated organic radical  $\text{Cl-R-Si(R') (R'')H}$  are practically unknown in the literature. Such compounds are of interest producing polymers having chains with alternating silicon atoms and hydrocarbons. In the present work the authors prepared chloromethyl-methylphenyl silane, chloromethyldiphenylsilane and chlorophenylmethylsilane by reducing the corresponding chlorosilanes with lithium aluminum hydride. In the case of chlorophenylmethylphenylchlorosilane the reduction proceeded smoothly and the yield of chlorophenylmethylphenylsilane was 69.5 - 72% irrespective of the order of addition of the reagents. The reduction of chloromethylmethylphenylchlorosilane proved more complex;

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D227/D302

Synthesis of some ...

when  $\text{LiAlH}_4$  was added to chlorosilane the yield of product was 83% and reduction of chlorine in the chloromethyl group was practically non-existent. When the order of addition was changed the yield of chloromethylmethylphenylsilane was only 56%. The most difficult reaction occurred in the case of chloromethyldiphenylsilane; under optimum conditions, the yield of chloromethyldiphenylsilane was only 67% and that of methyl-diphenylsilane 7%. Experimental procedure: The starting materials were prepared by reacting the corresponding dichlorosilane (chloromethylmethyl-dichlorosilane or chlorophenylmethyldichlorosilane) with phenylmagnesium bromide under the usual Grignard reaction conditions. The products of distillation were used in the reduction which was carried out in ether, adding the reducing agent to the chlorosilane. After completing the addition the mixture was refluxed for 6 hours, cooled and decomposed with 5% HCl. The ethereal solution was washed and distilled. Redistillation of the residue in the case of chloromethylmethylphenylchlorosilane reduction yielded chloromethylmethylphenylsilane b.pt. 99-100°C/14 mm  $n_D^{20}$  1.5326,

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D227/D302

Synthesis of some ...

$d_4^{20}$  1.0485  $MR_D$  50.49;  $MR_{calc}$  50.69. Chloromethyldiphenylsilane and chlorophenylmethylphenylsilane were also prepared; their properties are: b.pt. 175-180°C/15 mm  $n_D^{20}$  1.5842,  $d_4^{20}$  1.1089,  $MR_D$  70.25,  $MR_{calc}$  70.46; and b.pt. 172-180°C/25 mm  $n_D^{20}$  1.5795,  $d_4^{20}$  1.0982,  $MR_D$  70.50;  $MR_{calc}$  70.46, respectively. There are 7 references: 3 Soviet-bloc and 4 non-Soviet-bloc. The references to the English-language publications read as follows: H. Gilman, G. E. Dunn, J. Am. Chem. Soc. 73, 3404 (1951); U.S. Patent 2,527,591 (1950); G. Russell, J. Org. Chem. 21, 1190 (1950); R. A. Benkeser, D. Foster, J. Am. Chem. Soc. 74, 5314 (1952).

SUBMITTED: November 14, 1960

Card 3/3

15.817025480  
S/020/61/139/001/012/018  
B103/B226

AUTHORS: Andrianov, K. A., Corresponding Member AS USSR, Savushkina, V. I., Golubtsov, S. A., and Charskaya, B. A.

TITLE: Thermal condensation of dichloro silane with chlorobenzene

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 139, no. 1, 1961, 95 - 98

TEXT: The authors studied the thermal condensation of dichloro silane with chlorobenzene  $\text{H}_2\text{SiCl}_2 + \text{C}_6\text{H}_5\text{Cl} \rightarrow \text{C}_6\text{H}_5\text{SiHCl}_2 + \text{HCl}$  (1), 30 % phenyl dichloro silane resulting in the process. In addition to reaction (1), they determined the substitution of the second hydrogen atom at silicon by the phenyl group. In the presence of the high temperatures used here (640 - 700°C), substitution of the hydrogen atom at silicon by a chlorine atom was furthermore to be expected. As a result of this complicated process, the following compounds are present among the reaction products: Diphenyl dichloro silane and phenyl-trichloro silane (optimum total yield together with phenyl dichloro silane: 74.6 %); furthermore, benzene (3), (4), and trichloro silane (3). The present study proves that the yield of individual

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Thermal condensation of dichloro...

reaction products is, above all, dependent upon temperature. Up to about 640 - 660°C (optimum temperature of reaction (1)) the yield of phenyl dichloro silane increases up to 41.7 %, and, with a further temperature rise up to 700°C, it decreases to 12 %. The yield of phenyl trichloro silane increases at 640 - 660°C to 18.3 %, and up to 700°C continues increasing up to 26 %. The yield of diphenyl dichloro silane first increases (up to 12.4% at 660°C), at 700°C, however, decreases to 2.5 %. These facts speak in favor of a continuously increasing rate of the reaction mentioned at the beginning. For these reasons, silane and chlorosilane are practically entirely absent in the reaction products, and in the decomposition of dichloro silane neither hydrogen (2) nor side reactions of the chlorination of chlorosilane hydrides (3), (4) have been proved to develop. The authors consider it quite probable that part of phenyl trichloro silane forms according to the scheme  $\text{HSiCl}_3 + \text{C}_6\text{H}_5\text{Cl} \rightarrow \text{C}_6\text{H}_5\text{SiCl}_3 + \text{HCl}$  (5). The rate of reactions (3), (4), and (5):  $\text{C}_6\text{H}_5\text{SiHCl}_2 + \text{C}_6\text{H}_5\text{Cl} \rightarrow (\text{C}_6\text{H}_5)_2\text{SiCl}_2 + \text{HCl}$  (2);

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B103/B226

Thermal condensation of dichloro...

$\text{H}_2\text{SiCl}_2 + \text{C}_6\text{H}_5\text{Cl} \longrightarrow \text{HSiCl}_3 + \text{C}_6\text{H}_6$  (3);  $\text{C}_6\text{H}_5\text{SiHCl}_2 + \text{C}_6\text{H}_5\text{Cl} \longrightarrow \text{C}_6\text{H}_5\text{SiCl}_3 + \text{C}_6\text{H}_6$  (4) increases more considerably than that of (2). At 680°C the formation rates of phenyl trichloro silane tend toward similar values. Formation of trichloro silane and phenyl trichloro silane can hardly be explained other than by (3) and (4); i.e., neither by disproportionation:  $2\text{H}_2\text{SiCl}_2 \longrightarrow \text{HSiCl}_3 + \text{H}_3\text{SiCl}$  (6) nor by decomposition of dichloro silane:  $3\text{H}_2\text{SiCl}_2 \longrightarrow \text{Si} + 2\text{HSiCl}_3 + 2\text{H}_2$  (7). Also, the formation of benzene can be explained only by reactions (3) and (4), and not by pyrolysis of chlorobenzene in a reducing medium. In special experiments conducted on this pyrolysis, the authors found that the benzene yield did not exceed 9% (in hydrogen medium) and 2.2% (in silane medium). On the other hand, in the production of phenyl dichloro silane 55 - 60% benzene formed. Also the small yield of highly boiling products in the production of phenyl dichloro silane points to the unimportant part played by pyrolysis. S. A. Platonova and T. A. Klochova participated in the experimental part of the study. There are 3 figures, 3 tables, and 2 Soviet-bloc references.

SUBMITTED: March 22, 1961  
Card 3/3

S/062/62/G00/006/004/008  
B117/B101

AUTHORS: Morozova, L. P., Golubtsov, S. A., Andrianov, K. A., Trofimova, I. V., and Morozov, N. G.

TITLE: Formation of alkyl (aryl) chlorosilanes in direct reaction of alkyl (aryl) chlorides with silicon. Communication 1. Selectivity of silicon and copper catalysts, and formation of methyl dichlorosilane

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 6, 1962, 1005 - 1011

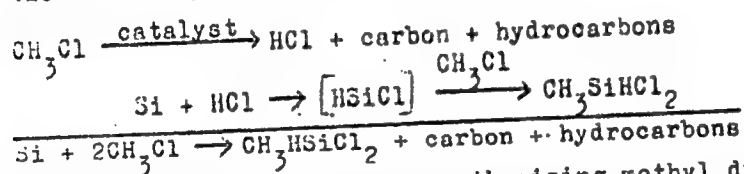
TEXT: Production conditions, precipitating agents, and promoters affecting the selective activity of silicon and copper hydroxide catalysts in the formation of methyl dichlorosilane and dimethyl dichlorosilane were studied. Sufficiently active catalysts can be obtained by using copper chloride and copper nitrate, but copper sulfate gives completely passive catalysts. Simultaneous precipitation of copper hydroxide and zinc hydroxide (~2% by weight) increases the selectivity of the catalyst. Sodium hydroxide (in the formation of methyl dichlorosilane) and  $\text{NH}_4\text{OH}$  or  $\text{Na}_2\text{CO}_3$  (in the formation of dimethyl dichlorosilane) are also studied.

Card 1/3

S/062/62/000/006/004/008  
B117/B101

Formation of alkyl ...

tion of dimethyl dichlorosilane) were found to be precipitating agents favoring the selectivity. The greatest effect on the selectivity of the catalyst is that exercised by promoters after the precipitation of hydroxides: Thus after introducing  $\text{ZnCl}_2$  the yield of dimethyl dichlorosilane reaches 60% and after introducing  $\text{Na}_2\text{SiO}_3$  that of methyl dichlorosilane reaches 45%. Thermal decomposition of methyl chloride on copper catalysts at 360-380°C (contact time 6-10 sec) was also studied. The hydrogen chloride separated in this reaction considerably affected the formation of methyl dichlorosilane. The following reaction course was suggested for the formation of methyl dichlorosilane:



The optimum temperature for synthesizing methyl dichlorosilane was found to be 350-380°C. At higher and lower temperatures, silicon tetrachloride,

Card 2/3

Formation of alkyl ...

S/062/62/000/006/004/008  
B117/B101

trichlorosilane, and methyl trichlorosilane were mainly formed. There are 4 tables.

SUBMITTED: December 9, 1961

Card 3/3

40912

S/191/62/000/010/005/010  
B101/B186

5.3833 ,

AUTHORS: Golubtsov, S. A., Popeleva, G. S., Andrianov, K. A., Zaslavskaya, N. I.

TITLE: Thermal condensation of trichloro silane and methyl dichloro silane with vinyl chloride

PERIODICAL: Plasticheskiye massy, no. 10, 1962, 21 - 27

TEXT: Thermal condensation of vinyl chloride with methyl dichloro silane into methyl vinyl dichloro silane (I), and vinyl chloride with trichloro silane into vinyl trichloro silane (II) was conducted in a reaction tube of stainless steel under atmospheric pressure and at 30 sec contact time. Optimum reaction conditions were determined, and the condensation mechanism and thermal decomposition were studied. Results: The optimum temperature for synthesizing (I) is 530 - 540°C, the yield is 75 - 80% as referred to the reacting methyl dichloro silane. The side products are methyl trichloro silane, butadiene, high-boiling products, and gases containing 2% H<sub>2</sub>, 5% C<sub>2</sub>H<sub>4</sub>, 4% C<sub>2</sub>H<sub>3</sub>Cl, 2% CH<sub>4</sub>, and up to 1% C<sub>2</sub>H<sub>5</sub>. The thermal decomposition of

Card 1/3



S/191/62/000/010/005/010  
B101/B186

## Thermal condensation ...

methyl dichloro silane starts in at 400°C and reaches 20% at 570°C. The gaseous decomposition products contain 75% H<sub>2</sub> and 25% CH<sub>4</sub>. The solid residue contains 65% Si but no free carbon. A radical mechanism is assumed, as the solid decomposition products catalyze the decomposition of methyl dichloro silane. The optimum conditions for the synthesis of II are: a molar ratio HSiCl<sub>3</sub> : C<sub>2</sub>H<sub>3</sub>Cl = 1 : 1.5 and 500 - 520°C. The yield is 70 - 75% as referred to the reacting HSiCl<sub>3</sub>, and 60% referred to the reacting vinyl chloride. If the ratio is reduced to 1 : 0.6, the temperature has to be raised to 560 - 580°C. A ratio of 1 : 1 yielded 80% II with respect to HSiCl<sub>3</sub>. The side products are equal amounts of SiCl<sub>4</sub> (15 g-moles per 100 g-moles HSiCl<sub>3</sub>) and high-boiling polymers. The gas contains 5% H<sub>2</sub>, 12% C<sub>2</sub>H<sub>4</sub>, 3% C<sub>2</sub>H<sub>6</sub>, the solid residue contains 10 - 50% Si and 30 - 75% elementary carbon. The thermal decomposition of HSiCl<sub>3</sub> in H<sub>2</sub>, SiCl<sub>4</sub>, and Si sets in at 560 - 570°C. The effect of the reaction vessel wall on the composition of the reaction products confirms the radical mechanism of the reaction. A surface increase of the reaction vessel by Raschig rings, Card 2/3

Thermal condensation ...

S/191/62/000/010/005/010  
B101/B186

increases the yield of  $\text{SiCl}_4$  and decreases the yield of II. Purification of the reaction vessel with alkali also reduces the yield of II. A nitrogen oxide addition of 2% reduces the yield of II to 43% and increases that of  $\text{SiCl}_4$  to 13%. In the reaction of II with HCl at 560 - 570°C, the gas consists of 93%  $\text{H}_2$  with 7%  $\text{C}_2\text{H}_4$ . There are 8 figures and 3 tables. The most important English-language references are: English Patent 752700 (1956), C. A., 51, 7402 (1957); US Patent 2770634 (1956), C. A., 51 10560 (1957), Japan Patent no. 16 (1951), C. A., 52, 3673 (1958).

Card 3/3

ANDRIANOV, K.A.; TURETSKAYA, R.A.; GOLUBTSOV, S.A.; TROFIMOVA, I.V.

Formation reactions of alkyl(aryl)chlorosilanes in the direct  
interaction of alkyl(aryl) chlorides with silicon. Report No. 12:  
Effect of hydrogen chloride on the formation of ethylchlorosilanes.  
Izv. AN SSSR.Otd.khim.nauk no.10:1788-1794 O '62. (MIRA 15:10)  
(Silane) (Hydrochloric acid)

33921

S/079/62/032/002/006/011  
D204/D303

5.3700

AUTHORS: Popeleva, G.S., Savushkina, V.I., Andrianov, K.A. and  
Golubtsov, S.A.

TITLE: Interaction of the halogen derivatives of aryl chlorosilanes  
with hydrogen chlorosilanes

PERIODICAL: Zhurnal obshchey khimii, v.32, no. 2, 1962, 557-562

TEXT: High temperature condensations of methyl dichlorosilane (I) with methyl chlorophenyl dichlorosilane (II) (reaction 1), methyl phenyl chlorosilane (III) with p-dichlorobenzene (reaction 2) and of III with methyl chlorophenyl phenyl chlorosilane (IV) (reaction 3) were investigated. Reaction 1 was carried out with 1:1 molar ratios of the reagents at 570, 600, 620, 640 and 670°C, with contact times of 40, 50, 60 and 80 sec., in stainless steel tubes and yielded a mixture of the ortho-, meta- and para-isomers of bis (methyl dichlorosilyl) benzene (A). It was found that the yield of A, under optimum conditions (640°C, 60 sec.), was 27%, calculated with respect to I. The product then consisted of 60% of the liquid meta-isomer and 40% of the crystalline ortho- and para-isomers. Reaction 2 at  
Card 1/2

33921

S/079/62/032/002/006/011

D204/D303

Interaction of the halogen ...

550°C, with a contact time of 40 sec., in silica tubes, gave IV in 34.6% yield, (calculated with respect to III), when the molar ratio of III to the p-dichlorobenzene was 2:1. Reaction 3 was carried out in silica tubes, at 650°C and with 40 sec. contact time, with reagents in 1:1 molar ratio, and gave para-bis (methyl phenyl chlorosilyl) benzene (B), in ~ 30% yield (calculated with respect to III). The structure of B was confirmed by a Grignard synthesis. Physical constants of the products and full experimental details are given. There are 2 figures, 4 tables and 15 references: 9 Soviet-bloc and 6 non-Soviet-bloc. The 4 most recent references to the English-language references read as follows: British Pat. 752,700 (1956); Ch.A., 51, 7402, (1957); Ch.A. 47, 3875, (1953); Ch.A. 47, 3334, (1953).

SUBMITTED: January 30, 1961

Card 2/2

S/079/62/032/003/004/007  
D204/D302

AUTHORS: Trofimova, I.V., Lobusevich, N.P., Golubtsov, S.A. and  
Andrianov, K.A.

TITLE: The effect of certain metallic additions to Si-Cu alloys  
on their activity in the reaction with methyl chloride

PERIODICAL: Zhurnal obshchey khimii, v. 32, no. 3, 1962, 841-846

TEXT: The optimum amount of Cu and the effect of adding metals usually present in Cu and Si on the synthesis of methyl chlorosilanes were investigated, at 350-370°C, under 4 atm, by a method described earlier. Purified Si (total Al+Ca+Fe+Ti < 0.2%) and Kp-1(Kr-1)Si were used, with M-1 and M-0 copper. Assessment of the additions of Al, Ca, Fe, Ti, Pb, Sb and Bi was made on the basis of the yield of methyl chlorosilanes (g/kg alloy/hr) and by the selective formation of  $\text{Me}_2\text{SiCl}_2$ . The optimum Cu content proved to be 7-10%. Using pure silicon, Al and Ti lowered the alloy activity when present to the extent of 0.2-0.3%, whilst Fe and Ca

Card 1/2

00513R000515920

38678

S/079/62/032/006/006/006  
D202/D304

5.2700

AUTHORS: Belyakova, Z. V., Golubtsov, S. A. and Yakusheva, T.M.

TITLE: Synthesis of organosilicon monomers containing the  
B-cyanoethyl group

PERIODICAL: Zhurnal obshchey khimii, v.32,no.6, 1962, 1997-2003

TEXT: The authors studied the cyanoethylation of methyldichlorosilane, but with no success. Syntheses were carried out of methyl, ethyl and phenyl derivatives of B-cyanoethyldichlorosilane from the trichlorocompound. Methyl-B-cyanoethyldichlorosilane was obtained from dimethylcadmium by the method of Cooper and Prober. The pure methyl compound was isolated from the reaction products by esterification with iso-butyl alcohol. Using ethyl and phenyl magnesium bromides, ethyl and phenyl-cyanoethyldichlorosilanes were prepared, the last not being previously described in literature. By a full or partial esterification of various cyanoethylchlorosilanes the authors obtained: B-cyanoethyltriethoxysilane, ethyl-B-cyanoethyldichlorosilane, B-cyanoethyltriacetoxysilane,

Card 1/2

Synthesis of organosilicon ...

S/079/62/032/006/006/006  
D202/D304

methyl-β-cyanoethyldiethoxychlorosilane, and 6 new compounds:  
β-cyanoethyldiethoxychlorosilane, β-cyanoethyldibutoxychlorosi-  
lane, β-cyanoethylbutoxydichlorosilane, methyl-β-cyanoerhyldibu-  
toxysilane, methyl-β-cyanoethyldi-iso-butoxysilane and ethyl-β-  
cyanoethyldiacetoxysilane.

SUBMITTED: June 30, 1961

Card 2/2



S/080/62/035/007/006/013  
D214/D307

AUTHORS: Turetskaya, T.A., Golubtsov, S.A., Tromimova, I.V.  
and Andrianov, K.A.

TITLE: The influence of additions of some metals on the  
activity of silicon-copper alloy in its reaction  
with ethyl chloride

PERIODICAL: Zhurnal prikladnoy khimii, v. 35, no. 7, 1962,  
1496-1502

TEXT: The general and selective activities of Si-Cu alloys  
in the reaction with EtCl to give a mixture of ethyl chlorosilanes  
are affected by the chemical nature of the alloy. The presence of  
1-2% Fe, Al, Ca or Ti in the alloy increases its general activity,  
while Al, Ca and Ti also increase its selective activity by increas-  
ing the yield of EtSiHCl<sub>2</sub>. The increase in activity is more evident  
at low Cu concentrations. The added metals are localized at the  
interphase boundaries in the alloy, these being the active centers  
in the reaction. The influence Ca bears on the activity of the

Card 1/2

The influence of additions ...

S/080/62/035/007/006/013  
D214/D307

of the alloy is affected by the presence of other metals. Fe in concentrations of up to 10%, does not influence the process. Concentrations of Bi and Sb of the order of 0.001% influence the selective activity and increase the yield of  $\text{Et}_2\text{SiCl}_2$ . Pb, in these concentrations, acts as a catalytic poison. At higher concentrations, both Bi and Sb also become poisons and at concentrations of 0.01% these metals render the alloy inactive. The mechanism of the action of the added metals cannot as yet be explained. There are 4 figures and 6 tables. ✓

SUBMITTED: December 9, 1960

Card 2/2

5/004 of 000/001/002/007  
2001 215

**AUTHORS:** Turetskaya, R. A., Golubtsov, S. A., Andrianov, K. A.,  
Tsvanger, T. A., Prigozhin, B. Yu.

**TITLE:** Direct synthesis of ethyl chlorosilanes

**PERIODICAL:** Khimicheskaya promyshlennost', no. 1, 1963, 18 - 20

**TEXT:** A method of directly synthesizing ethyl chlorosilanes in a fluidized bed at 360 - 380°C, wherein ethyl chloride is caused to react with a copper-silicon alloy was described by these authors in a series of previous studies (DAN SSSR, 196, 4 - 1962, 1963, 1964, 1965, 1966, 1967; Zh. tekhn. Khim., no. 1, 1963, 1964, 1965, 1966, 1967). For comparison, data of lab tests and experiments in a pilot plant with a reaction vessel (of 300 mm diameter and a charge capacity of 250 kg alloy) are given in the present paper. The above data were found to be consistent except for the ethyl chloride and yield per unit kg of contact mass, this being 270 - 700 g in lab tests and 10 - 15 kg in the pilot plant. The difference is due to the longer contact time (approximately 10 min in the pilot plant). The composition of the resulting mixture of ethyl chlorosilanes is given in the following sequence: head fraction;

Direct synthesis of ethyl ...

S/064/63/000/001/002/007  
B1 1 B186

$\text{C}_2\text{H}_5\text{SiHCl}_2$ ;  $\text{C}_2\text{H}_5\text{SiCl}_3$ ;  $(\text{C}_2\text{H}_5)_2\text{SiHCl}$ ;  $(\text{C}_2\text{H}_5)_3\text{SiCl}$ , and residue. The data of lab tests in method A are the following, contact mass of Cu-Si alloy containing 20 % Si: 0, 27, 20, 7, 37, 9; method B, addition of 27 - 28 % by volume of  $\text{H}_2$  during the experiment: 3, 41, 16, 11, 18, 10; method C, addition of 20 - 23.5 % by volume of  $\text{HCl}$  during the experiment: 11, 49, 16, 4, 16, 6; method D, addition of alloy during the experiment: 2, 11, 18, 5, 16, 9, and method E contact mass of Cu-Si alloy containing 20 % Si promoted by 0.003 t of Sb: 11, 18, 16, 4, 16, 6. For the pilot experiments, these data are 1, 27, 37, 0, 0, 0 for method A, 11, 18, 16, 4, 16, 6 for method B, 3, 46, 27, 0, 17, 7 for method C, 3, 36, 20, 7, 20, 11 for method D, and 4, 20, 28, 0, 16, 1 for method E. The alloy promoted by Sb showed an increase in selectivity for the ethyl siloxane yield, whereas the ethyl siloxane yield was 17 % for the alloy promoted by  $\text{H}_2$  addition during the experiment. The yield of ethyl siloxane from ethylene and ethane by the reaction of ethyl chloride. The synthesis of polyethyl siloxanes from ethyl chlorosilanes is compared with that from ethyl ethoxysilanes. In the first case, 4.2 t and in the second case 10.2 t of raw material is required per ton of polyethyl siloxane. The apparatus per unit volume, calculated for the first case is 1.2 t.

Direct synthesis of ethyl ...

S/064/63/000/001/002/007  
B101, B166

... in the first case and 2.4 g/hr-l in the second case. Further improvements are possible by increasing the yield of diethyl ischloro-  
allene and by complete inhibition of the formation of dehydrochlorination  
products. There are 2 figures and 4 tables.

Card 3/3

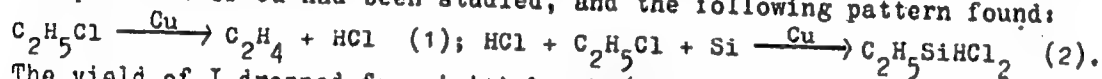
S/062/63/000/001/012/025  
B101/B186

AUTHORS: Golubtsov, S. A., Turetskaya, R. A., Andrianov, K. A., and  
Vabel', Ya. I. (Deceased)

TITLE: Formation of alkyl (aryl) chloro silanes by direct reaction of  
an alkyl (aryl) chloride with silicon. Communication 3. Direct  
synthesis of ethyl dichloro silane

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk,  
no. 1, 1963, 87 - 90

TEXT: In a previous paper (Izv. AN SSSR, Otd. khim. n. 1962, 1788) the syn-  
thesis of ethyl dichloro silane (I) by reaction of ethyl chloride with Si in  
the presence of Cu had been studied, and the following pattern found:



The yield of I dropped from initial ~75% after 30 min to a constant level  
of 15-25% due to the decreasing rate of reaction (1). The objective of the  
present study was an increase in the yield of I by adding Cu-Si alloy during  
the reaction. The synthesis was carried out in a fluidized bed of the Cu-Si  
Card 1/2

Formation of alkyl ...

S/062/63/000/001/012/025  
B101/B186

alloy containing 17% Cu at 370°C. Results: After 3 hrs reaction, the content of I in the reaction product was 12-19%; the other components of the reaction product were ethyl trichloro silane and diethyl dichloro silane. 20 min after adding 13% fresh Cu-Si alloy, the content of I rose to 51.5 - 59%, the rate of formation of ethyl chloro silanes had increased to the 2.5-fold. In a continuous experiment with supply of Cu-Si every 30 min, the process was stabilized after 18 hrs. The Cu content in the contact mass rose from 17 to about 35%, the content of I from 15-23 to 50-55%, and the formation of ethyl chloro silanes from 418 g/hr to 725 g/hr per kg of alloy. These values remained constant for 240 hrs. There are 3 figures and 2 tables.

SUBMITTED: April 25, 1962

Card 2/2

L 10595-63

ENP(j)/EPF(c)/EWT(m)/BDS ASD Pc-L/Pr-L RM/WW

ACCESSION NR: AP3000943

S/0064/63/000/003/0011/0018

65

AUTHOR: Turetskaya, R. A.; Golubtsov, S. A.; Trofimova, I. V.; Andrianov, K. A.

TITLE: The influence of some kinetic and hydrodynamic conditions on the direct synthesis of ethylchlorosilanes

SOURCE: Khimicheskaya promyshlennost', no. 3, 1963, 11-18

TOPIC TAGS: Cu-Si alloy, kinetic conditions, hydrodynamic conditions, ethylchlorosilane

ABSTRACT: The optimum particle size of Cu-Si alloy (75-250 microns) for the direct synthesis of ethylchlorosilanes, and the critical rate of fluidizing the alloy in the stream of ethyl chloride in reactors of 20-100 mm diameter, were determined. Investigation of reagent contact time, in intervals from 0.3-6.0 sec., on the course of reaction showed composition of reaction products was practically independent of contact time. Optimum synthesis temperature was 360-380C (300-390 degrees range investigated). Change in properties of catalyst and its effect on reaction with ethyl chloride was investigated. Orig. art. has 16 figures.

ASSOCIATION: none

Card 1/2/



TURETSKAYA, R. A.; GOLUBTSOV, S. A.; TROFIMOVA, I. V.; ANDRIANOV, K. A.

Effect of some kinetic and hydrodynamic conditions on the  
direct synthesis of ethylchlorosilanes. Khim. prom. no.3:  
171-178 Mr '63. (MIRA 16:4)

(Silane)

LOBUSEVICH, N.P.; LAYNER, D.I.; TROFIMOVA, I.V.; MALYSHEVA, L.A.;  
ANDRIANOV, K.A.; GOLUBTSOV, S.A.

Reactions of alkyl (aryl) chlorosilane formation by the direct  
interaction between alkyl (aryl) chlorides and silicon. Report No.5:  
Phase composition of silicon-copper contact masses in reactions with  
methyl chloride. Izv. AN SSSR Ser.khim. no.10:1757-1766 0 '63.  
(MIRA 17:3)

1. Nauchno-issledovatel'skiy i proyektnyy institut splavov i  
obrabotki tsvetnykh metallov.

POPELEVA, G.S.; ANDRIANOV, K.A.; GOLUBTSOV, S.A.; POPKOV, K.K.

Thermal addition of hydrochlorosilanes to alkenylchlorosilanes.  
Izv. AN SSSR. Ser. khim. no.11:2041-2042 N '63. (MIRA 17:1)

1 3/312-62 EWP(j)/EPF(c)/EWT(m)/BDS ASE 1-1-1-1 RM/AM  
AP300227

Authors: Turetskaya, R. A.; Trofimova, I. V.; Andrianov, K. A.; Golubtsov, S.

Topic: The question of the role of the phase structure of silicon-copper contact masses in the direct synthesis of ethylchlorosilanes

SOURCE: Zhurnal obshchey khimii, v. 33, no. 6, 1963, 2015-2018

65  
64

TOPIC TAGS: phase structure, silicon-copper, synthesis, ethylchlorosilane, Cu sub 3 Si, silane, catalyst, ethyl chloride, dehydrochlorination, ethyldichlorosilane, diethyldichlorosilane

ABSTRACT: According to data obtained as well as literature, the role of the intermetallic Cu sub 3 Si compound, which disintegrates because of Si reacting with ethylchloride and which is regenerated in the process, consists in the formation of silanes and partial renewal (of the catalyst). The concentration of Cu sub 3 Si does not determine selective activity of catalyst activity in ethylchloride reaction; product yields are essentially the same whether contact is made with Cu sub 3 Si, or just a mixture of Cu and Si (pure or with additives). If there is no Cu sub 3 Si, Cu provides the catalytic activity; the generation

Card 1/2

10019-43

10019-43 02627

Chlorosilane (from ethyl chloride) on contact with Ru-catalyst is almost  
inactive, dropping notably with time. This is due to catalytic activity  
degradation. Information is assumed to determine the relative formation of  
chlorosilane (in preference to diethyldichlorosilane) in the direct  
hydrochlorination process. Orig art. has: 1 figure, 2 tables

ASSOCIATION: None

SUBMITTED: 26Apr62

DATE ACQ: 20Jul63

ENCL: 00

SUB CODE: 00

NO REF SOV: 010

OTHER: 001

Card 2/2

LAYNER, D.I.; MALYSHEVA, L.A.; YEMEL'YANOV, L.G.; TROFIMOVA, I.V.;  
LOBUSEVICH, N.P.; GOLUBTSOV, S.A.

Rate of cooling silicon-copper alloys. TSvet. mat. 36 no.8;  
76-79 Ag '63. (MIRA 16:9)  
(Silicon-copper alloys—Metallography)  
(Nonferrous ingots—Cooling)

L 18899-63.

ENP(j)/EPF(c)/EWT(m)/BDS ASD Pr-l/Pc-l RM/WW/MAY  
ACCESSION NR: AP3006593 3/0020/63/151/006/1329/1331

AUTHORS: Golubtsov, S. A.; Andrianov, K. A. (Corr. member AN SSSR); Turetskaya, P. A.; Belikova, Z. V.; Trofimova, I. V.; Morozov, N. G. 69

TITLE: Reaction mechanism in the formation of dialkyldichlorosilanes

SOURCE: AN SSSR. Doklady\*, v. 151, no. 6, 1963, 1329-1331 7

TOPIC TAGS: dialkyldichlorosilane, dichlorosilane, silane, silicon chloride, copper chloride, hydration, methyl chloride, alkyl chloride

ABSTRACT: Authors showed that during the interaction of alkyl chloride with silica in the presence of copper, dialkyldichlorosilanes are formed. Copper monochloride, which is formed during the reaction of methyl chloride with copper, plays an important part in the synthesis of dialkyldichlorosilanes. The process consists of the adsorption of alkylchloride and its interaction with copper forming CuCl. Copper monochloride reacts with silica forming an intermediate product SiCl<sub>2</sub>. The removal of CuCl from the reaction zone by means of hydration with hydrogen, results in the discontinuation of

Card 1/2

L 18899-63

ACCESSION NR: AP3006593

diethyldichlorosilane formation. Orig. art. has: 1 table, 1 figure  
and 6 formulas.

ASSOCIATION: none

SUBMITTED: 09Apr63

DATE ACQ: 27Sep63

ENCL: 00

SUB CODE: CH

NO REF SOV: 005

OTHER: 001

Card 2/2



ACCESSION NR: AP4018162

S/0191/64/000/003/0022/0024

AUTHORS: Lobusevich, N.P.; Trofimova, I.V.; Andrianov, K.A.;  
Golubtsov, S.A.

TITLE: Effect of metal halides on the activity of silicon-copper  
alloys in the synthesis of methylchlorosilanes.

SOURCE: Plasticheskiye massy\*, no.3, 1964, 22-24

TOPIC TAGS: silicon copper catalyst, catalyst activity, methylchlorosilane synthesis, dimethyldichlorosilane synthesis, cuprous chloride, zinc chloride, silicon copper alloys, sodium halide, catalyst activator, metal halides

ABSTRACT: Activation of silicon-copper alloys containing 20% silicon with 3-7% CuCl increases the dimethyldichlorosilane content in the mixture of methylchlorosilanes by 10-20% in reactions at 4.5-5 atmospheres pressure. (no favorable results at atmospheric pressure); the optimum temperature is 3600. ZnCl<sub>2</sub> appears to be a more effective activator than CuCl since its introduction increases the dimethyldi-

Card 1/2

ACCESSION NR: AP4018162

chlorosilane content by 15-30% and the general activity of the silicon-copper alloy by 1.5 to 2 times. The optimum  $ZnCl_2$  concentration depends on the synthesis conditions, e.g., at atmospheric pressure, 370C, and 3-5%  $ZnCl_2$ , the dimethyldichlorosilane yield is 65-67%; at 3 atmospheres, 290C, and 1%  $ZnCl_2$  maximum yield was realized. The addition of 2-4% NaCl or NaF does not increase the yield of trimethylchlorosilane, but it does increase the yield of methyldichlorosilane from 5% to 12-20%. Orig. art. has: 4 tables and 5 figures.

ASSOCIATION: None

SUBMITTED: 00

SUB CODE: CH

DATE ACQ: 27Mar64

NR REF SOV: 001

ENCL: 00

OTHER: 010

ACCESSION NR: AP4034567

S/0079/64/034/004/1111/1113

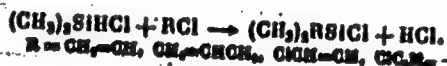
AUTHOR: Popeleva, G. S.; Andrianov, K. A.; Larionova, A. A.; Golubtsov, S. A.

TITLE: Thermal condensation of dimethylchlorosilane with certain organic chloro-derivatives.

SOURCE: Zhurnal obshchey khimii, v. 34, no. 4, 1964, 1111-1113

TOPIC TAGS: dimethylchlorosilane, thermal condensation, dimethylvinylchlorosilane, dimethylallylchlorosilane,  $\beta$  chlorovinyl dimethylchlorosilane, bis dimethylchlorosilyl ethylene, p chlorophenyl dimethylchlorosilane, disproportionation, monofunctional derivative, polyfunctional derivative, distillation, purification, etherification

ABSTRACT: This is a continuation of earlier investigations of the thermal condensation of chlorosilanes with different chloro-organic compounds. In this investigation the thermal condensation (at 500-550 C) of chloroorganics with dimethylchlorosilane were studied:



Card 1/2

ACCESSION NR: AP4034567

Dimethylvinylchlorosilane, dimethylallylchlorosilane, and compounds not described in the literature, beta-chlorovinyl dimethylchlorosilane, bis(dimethylchlorosilyl)ethylene, and p-chlorophenyldimethylchlorosilane were prepared by this method. In the high temperature condensation process disproportionation of the dimethylchlorosilane takes place with the formation of dimethyldichloro-, methyl-dichloro- and trichlorosilanes, which in turn condense with the chloroorganics to form di- and tri-functional compounds whose boiling points are near those of the desired monofunctional compounds. These cannot be separated even by repeated distillation. It was found the monofunctional compounds may be purified by partial etherification of the polyfunctional impurities with isobutyl alcohol. Orig. art. has: 1 table.

ASSOCIATION: None

SUBMITTED: 19Jan63

DATE ACQ: 11May64

ENCL: 00

SUB CODE: 00

NO REF BOV: 006

OTHER: 005

Card 2/2

BELYAKOVA, Z.V.; YAKUSHEVA, T.M.; GOLUBTSOV, S.A.

Reaction mechanism of addition of hydrides of chlorosilanes  
to acrylonitrile, Zhur. ob. khim. 3/4 no. 5:1480-1484 My '64.  
(MIRA 17:7)

**"APPROVED FOR RELEASE: 06/13/2000**

**CIA-RDP86-00513R000515920010-8**

**APPROVED FOR RELEASE: 06/13/2000**

**CIA-RDP86-00513R000515920010-8"**

"APPROVED FOR RELEASE: 06/13/2000

CIA-RDP86-00513R000515920010-8

APPROVED FOR RELEASE: 06/13/2000

CIA-RDP86-00513R000515920010-8"

LOBUSEVICH, N.P.; TROFIMOVA, I.V.; ANDRIANOV, K.A.; GOLUETSOV, S.A.

Effect of moisture, methanol, and oxygen in methyl chloride  
on the synthesis of methylchlorosilanes. Zhur.prikl. khim.  
37 no. 5:1148-1152 My '64. (MIRA 17:7)



**"APPROVED FOR RELEASE: 06/13/2000**

**CIA-RDP86-00513R000515920010-8**

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**APPROVED FOR RELEASE: 06/13/2000**

**CIA-RDP86-00513R000515920010-8"**

CuCl = Si showed that even at low temperatures (1800), the ratios of peak  
in the mass spectrum correspond to the ratios of the

"APPROVED FOR RELEASE: 06/13/2000

CIA-RDP86-00513R000515920010-8

pyridinevinylchlorosilane in accordance with the following formulas:

APPROVED FOR RELEASE: 06/13/2000

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CIA-RDP86-00513R000515920010-8

APPROVED FOR RELEASE: 06/13/2000

CIA-RDP86-00513R000515920010-8"

...acting the chlorosilanes with perfluoro-1,1-dihydrobutyl acetate in the presence of

COLUBISOV, S.A.; BELYAKOVA, Z.V.; POMERANTSEVA, M.G.

Cleavage of siloxanes by silicon tetrachloride. Zhur. ob. khim.  
35 no.6:1044-1048 Je '65.

Reaction of silane hydrides with allyl chloride. Ibid.:1048-1052  
(MIRA 18:6)



BELYAKOVA, Z.V.; GOLUBTSOV, S.A.; YAKUSHEVA, T.M.

Addition of trichlorosilane to acrylonitrile and allyl cyanide.  
Zhur. ob. khim. 35 no.7:1183-1186 J1 '65. (MIRA 18:8)

L 1255-66 EPF(c)/EWP(j)/EWT(m)/T RM

ACCESSION NR: AP5021674

AUTHOR: Lobusevich, N. P.; Trofimova, I. V.; Andrianov, K. A.; Golubtsov, S. A. 44.55 44.55 44.55 40

UR/0080/65/038/008/1887/1889

547.211'222'245

TITLE: Effect of methyl chloride and vinyl chloride on the synthesis of methyl-chlorosilanes 44.55

SOURCE: Zhurnal prikladnoy khimii, v. 38, no. 8, 1965, 1887-1889

TOPIC TAGS: chloride, silane, methylene chloride, vinyl chloride, catalysis, copper, silicon, aluminum, antimony

ABSTRACT: Methyl chloride obtained by chlorination of natural gas contains up to 1.7 vol. % methylene chloride and 0.2-3.0% vinyl chloride. It is known that at temperatures of 300-350C methylene chloride reacts with silicon copper catalysts with formation of hexachlorodisilane methane and also of hydrogen containing chlorosilanes. Under the conditions of the reaction of methyl chloride with silicon-copper catalysts, the methylene chloride can react with the silicon with information of analogous compounds, and can undergo decomposition with the formation of carbon, which deactivates the catalyst. Carbonization of the catalyst was observed even after short term synthesis, with the introduction of

Card 1/2

L 1255-66

ACCESSION NR: AP5021674

6-7% of methylene chloride into the methyl chloride. In experiments in a pressurized fluidized bed on an alloy promoted with antimony, an investigation was made of the effect of vinyl chloride, whose concentration in the mixture with methyl chloride was varied from 0.16 to 4.0 vol. %. No adverse effect on the process was observed at concentrations up to 0.2%. In the reaction of methyl chloride with an alloy of the composition  $\text{Cu}_3\text{Si}$ , vinyl chloride in concentrations higher than 0.16% sharply lowers overall activity and slightly lowers selective activity. For  $\text{Cu}_3\text{Si}$  alloys and mixtures of copper and silicon powders with addition of 0.5% aluminum, the introduction of more than 0.16% vinyl chloride causes a greater decrease in overall activity than for catalysts with an antimony additive. In this case, large amounts of still residues are formed (15-40%). In general, it is concluded that under the conditions of the synthesis, vinyl chloride reacts with silicon with the formation of vinyl trichlorosilane, ethyl dichlorosilane, and dimethyl vinyl chlorosilane, and that this inhibits the separation of dimethylchlorosilane from the mixture of methylchlorosilanes. Orig. art. has: 3 figures and 1 table

SUBMITTED: 17Jun63

ENCL: 00

SUB CODE: MM, GC

NR REF SOV: 003

OTHER: 002

Card 2/2 KC

BYKOVCHENKO, V.G.; ERMANSON, L.V.; GOLUBTSOV, S.A.

Effect of inhibitors on the reaction of trichlorosilane with  
chlorobenzene. Zhur. fiz. khim. 39 no.2:450-451 F '65. (MIRA 18:4)

SHAPATIN, A.S.; GOLUBTSOV, S. I.; SOLOV'YEV, A.A.; ZHIGACH, A.F.;  
SIRYATSKAYA, V.N.

Addition of silicon chloride hydrides to alkenyl carboranes.  
Plast. massy no. 12:19-21 '65 (MIRA 19:1)

L 15790-66 EWT(m)/EWP(j) RM  
ACC NR: AP6002225

SOURCE CODE: UR/0080/65/038/012/2882/2885

AUTHOR: Lobusevich, N. P.; Trofimova, I. V.; Andrianov, K. A.; Golubtsov, S. A.

ORG: none

TITLE: Effect of dimethyl ether, carbon dioxide, and carbon monoxide on the synthesis of methylchlorosilanes 44166 <sup>25</sup><sub>P</sub>

SOURCE: Zhurnal prikladnoy khimii, v. 38, no. 12, 1965, 2882-2885

TOPIC TAGS: carbon monoxide, copper containing alloy, carbon dioxide

ABSTRACT: The effect of dimethyl ether in the reaction between methyl chloride with silicon alloys containing 20% Cu and 10% Cu, respectively, activated by 0.002-0.004% Sb in the boiling layer at atmospheric and higher pressures was studied. Carbon dioxide and carbon monoxide (0.5-14.5%) were studied in the same reaction at atmospheric pressure using various contact masses. It was found that dimethyl ether, carbon monoxide and, under certain conditions, carbon dioxide are contact inhibitors of the reaction which produces methylchlorosilanes. The inhibiting effect of carbon dioxide and dimethyl ether is attributed to carbon monoxide which causes ir-

UDC: 547.211'222'245

Card 1/2

L 15790-66  
ACC NR: AP6002225

reversible pitting of the copper catalyst and also prevents the decomposition of the intermetallic compound  $\text{Cu}_3\text{Si}$  with the formation of catalytically active copper. The presence of less than 1%  $\text{CO}$ ,  $\text{CO}_2$  and  $\text{CH}_3\text{OCH}_3$  sharply reduces the rate of the reaction which produces methylchlorosilanes. Orig. art. has: 6 figures, 2 tables.

SUB CODE: 07/      SUBM DATE: 20Jun63/      ORIG REF: 003/      OTH REF: 004

Card 2/2 *mgs*

L 16200-66 ENT(m)/ENP(j)/T RM

ACC NR: AP6002226

SOURCE CODE: UR/0080/65/038/012/2886/2887

AUTHOR: Lobusevich, N. P.; Trofimova, I. V.; Andrianov, K. A.; Golubtsov, S. A.

ORG: none

TITLE: Chemisorptive action of impurities and the effect of chlorosilanes and methylchlorosilanes

SOURCE: Zhurnal prikladnoy khimii, v. 38, no. 12, 1965, 2886-2887

TOPIC TAGS: chemisorption, chlorosilane, copper containing alloy, silicon containing alloy

ABSTRACT: The chemisorptive mechanism of action of the impurities is experimentally confirmed by introducing reaction products, chlorosilanes and methylchlorosilanes, into methyl chloride. It has previously been noted that the mechanism of action of the impurities is associated with their adsorption on the active centers and with the pitting of the copper catalyst. The introduction of from 0.5 to 2.0% of chlorosilanes or methylchlorosilanes into methyl chloride results in a two to three-fold increase in productivity and an increase of dimethyldichlorosilane in the mix-

UDC: 661.723-13

Card 1/2



L 16200-66

ACC NR: AP6002226

ture. The different effects of reaction products on the interaction of the alloys with pure and technical methyl chloride is apparently associated with the selective adsorption of impurities. The introduction of insignificant amounts of reaction products into methyl chloride and the preliminary treatment of the alloys with chlorosilanes or methyl chlorosilanes result in their selective adsorption on the catalyst which prevents pitting of the catalyst by harmful impurities and improves the indicators of the process. It is shown that the activity of the reaction products from methyl chloride and silicon in preventing the harmful effect of impurities increases in the series:  $\text{HSiCl}_3 \geq \text{SiCl}_4 > \text{CH}_3\text{SiCl}_3 > (\text{CH}_3)_3\text{SiCl} > (\text{CH}_3)_2\text{SiCl}_2 > \text{CH}_3\text{HSiCl}_2$ . Orig. art. has: 2 tables.

SUB CODE: 07/

SUBM DATE: 09Jul63/

ORIG REF: 001/

OTH REF: 000

L 11,612-66 EWT(m)/T/EXP(1) WW/JW/RM  
ACC NR: AP6001497 (A)

SOURCE CODE: UR/0191/65/000/012/C319/0021

AUTHORS: Shapatin, A. S.; Golubtsov, S. A.; Solov'yev, A. A.; Zhigach, A. F.; Siryatskaya, V. N. 37

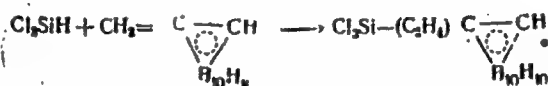
ORG: none

TITLE: Addition of hydrides of silicon chlorides to alkenyl carboranes 7.44.55 B

SOURCE: Plasticheskiye massy, no. 12, 1965, 19-21

TOPIC TAGS: silane, organic synthetic process, catalysis, silicon compound, catalyst, ferric chloride

ABSTRACT: A simplified method for synthesizing carborane siliconorganic monomers is offered. It consists of adding chlorosilicon hydrides to alkenyl carboranes, according to the equation: 7



The following reactions were studied: methylchlorosilane with carborane derivatives containing vinyl, isopropenyl, propenyl, or butenyl groups; trichlorosilane and dimethyl chlorosilane with vinyl and isopropenyl carborane; ethyl dichlorosilane and phenyldichlorosilane with isopropenylcarborane. Elementary analysis and

Card 1/2

UDC: 678.84

2

L 11612-66

ACC NR: AP6001497

physical properties of the resulting 10 compounds are reported. In the absence of the catalyst the reaction occurs only above 200C and results in very low yields. The yields increase to 80% and more, and the required temperatures are lowered by the addition of chloroplatinic acid or ferric chloride as catalysts. Orig. art. has: 2 tables and 1 equation.

SUB CODE: 07// SUBM DATE: none/ ORIG REF: 001/ OTH REF: 004

Card 2/2

L 13901-66 EWT(m)/EWP(j) RM

ACC NR: AP6002863 SOURCE CODE: UR/0286/65/000/024/0020/0020

INVENTOR: Popov, A. F.; Korneyev, N. N.; Golubtsov, S. A.;  
Popelava, P. S.

ORG: none

TITLE: Preparative method for bis(dimethylchlorosilyl)benzene.  
 Class 12, No. 176892/5

SOURCE: Byulleten' izobreteniy i tovarnykh znakov, no. 24, 1965, 20

TOPIC TAGS: silane

ABSTRACT: An Author Certificate has been issued for a preparative method for bis(dimethylchlorosilyl)benzene, involving the reaction of metallic magnesium with p-dibromobenzene and dimethyldichlorosilane. To simplify the process, it is carried out in the presence of 0.001—0.01 g-mol titanium tetrachloride catalyst/mol metallic magnesium.

SUB CODE: 07/ SUBM DATE: 22Jul64/ ATD PRESS: 419/

UDC: 547.419.5.07

Card 1/1

21  
B  
744/53

[SH]

L 20977-66 ENT(m)/EMP(j) RM

ACCESSION NR: AP5021673

UR/0080/65/038/008/1884/1886  
547.222

AUTHOR: Lobusevich, N. P.; Trofimova, I. V.; Andrianov, K. A.; Golubtsov,  
S. A.

TITLE: Effect of sulfur dioxide on the synthesis of methylchlorosilanes

SOURCE: Zhurnal prikladnoy khimii, v. 38, no. 8, 1965, 1284-1886

TOPIC TAGS: silane, catalysis, sulfur compound, silicate, copper, silicon,  
aluminum, antimony, chloride

ABSTRACT: The effect of sulfur dioxide was evaluated with respect to the yield and the content of dimethylchlorosilane in the mixture. With a content of sulfur dioxide greater than 0.002% in methyl chloride, there is a decrease in the overall activity of copper silicate promoted with antimony. A decrease in selective activity in the synthesis of dimethylchlorosilane is observed with an increase in concentration of sulfur dioxide from 0.002 to 0.01% and at concentrations from 0.01 to 1.0% the content of dimethylchlorosilane is practically unchanged. Selective activity of alloys with the composition Cu<sub>3</sub>Si(eta phase) in the absence of a promoter, as well as of mixtures of copper and silicon powders, decreases more rapidly than the activity of analogous alloys containing 0.005% antimony.

Card 1/2

L 20377-56

ACCESSION NR: AP5021673

With an increase in concentration of sulfur dioxide up to 2%, the synthesis of methylchlorosilanes over all the above catalysts stops. A particularly strong effect of sulfur dioxide is observed on the activity of alloys with increased content of aluminum (1% in an alloy with silicon and 87% copper). At sulfur dioxide concentrations of 0.002% the synthesis ceases. Mixtures of copper, silicon, and aluminum powders have a satisfactory and stable overall activity, but the selective activity decreases. With an increase in titanium content (0.5%) in alloys or in mixtures of copper and silicon powders, the introduction of sulfur dioxide into the methyl chloride leads to a decrease in activity and to a sharp increase in content of high melting products (up to 40% of the weight of the methylchlorosilane mixture). It was found that with an increase in reaction time of methyl chloride with a mixture of copper and silicon powders in the presence of 0.8% sulfur dioxide, the poisoning effect of the latter becomes stronger. Orig. art has: 5 figures and 1 table

ASSOCIATION: None

SUBMITTED: 17Jun63

NR REF SOV: 001

ENCL: 00

OTHER: 000

SUB\_CODE: FM, IC

Card 2/2 *MGS*

L 23717-66 EWT(m)/EWP(j)/T RM

ACC NR: AP6007118

SOURCE CODE: UR/0079/66/036/002/0345/0347

AUTHOR: Lobusevich, N. P.; Golubtsov, S. A.; Layner, D. I.; Malysheva, L. A.; Trofimova, I. V.

ORG: none

TITLE: On the problem of promoters and poisons in the direct synthesis of methylchlorosilanes

SOURCE: Zhurnal obshchey khimii, v. 36, no. 2, 1966, 345-347

TOPIC TAGS: silane, bismuth, phosphorus, antimony, copper alloy, silicon alloy, zinc, *chemical decomposition*

ABSTRACT: The kinetics of the decomposition of  $\text{Cu}_3\text{Si}$  were studied during its reaction with methyl chloride in the presence of promoters (arsenic, phosphorus mixed with antimony and zinc) and contact poisons (bismuth and phosphorus). Addition of the most active promoters lowers the temperature at which the  $\text{Cu}_3\text{Si}$  alloy begins to react with methyl chloride from  $330^\circ$  to  $270^\circ\text{C}$  in the case of arsenic and from  $330^\circ$  to  $290^\circ\text{C}$  in the case of the phosphorus-antimony mixture. The activation energy of the reaction between  $\text{Cu}_3\text{Si}$  and methyl chloride decreases by one-half when these promoters are introduced. The action of the zinc promoter increases the reaction rate, but the activation energy remains practically unchanged. Apparently, elemental zinc converts into zinc chloride which accelerates the reaction of dimethyldichlorosilane formation. Ad-

Card 1/2

L 23717-66

ACC NR: AP6007118

dition of bismuth or phosphorus sharply inhibit the reaction of  $\text{Cu}_3\text{Si}$  with methyl chloride even at high temperatures ( $390^\circ\text{C}$ ). Orig. art. has: 1 figure.

SUB CODE: 07/

SUM DATE: 01Dec64/

ORIG REF: 003/

OTH REF: 000

Card 2/2 *HW*





L 23834-66

ACC NR: AP6007125

The course of reaction (2), characteristic of compounds containing chlorine in the  $\beta$  position relative to silicon, demonstrates the structure of compound (III). The presence of chlorine in (III) in the  $\beta$  position relative to phosphorus indicates in turn that the addition of the chloride (I) to the alkenylsilane (II) follows Markovnikov's rule. The authors thank B. I. Ionin for his participation in a discussion of the work. Orig. art. has: 2 formulas.

SUB CODE: 07/

SUBM DATE: 04Mar65/

ORIG REF: 001/

OTH REF: 002

Card 2/2 *fv*

L 39718-66 EWT(m)/EWP(j)/T RM/GD-2

ACC NR: AP6007969

(A)

SOURCE CODE: UR/0191/66/000/003/0036/0037

AUTHOR: Turetskaya, R. A.; Golubtsov, S. A.; Davonar', V. G.

ORG: none

TITLE: Synthesis of triphenylchlorosilane from silicon tetrachloride and phenylsodiumSOURCE: Plasticheskiye massy, no. 3, 1966, 36-37

TOPIC TAGS: organic synthetic process, silicon compound, organosilicon compound

ABSTRACT: Tetraphenylsilane was prepared from silicon tetrachloride and phenylsodium by the known reaction (Polis, Ber. 18, 1514, 1885). The authors studied the possibility of preparing triphenylchlorosilane from these reagents. By a thorough purification of benzene chloride and the solvent (by a treatment with calcium hydride, phosphorus pentoxide, and subsequently with  $H_2SO_4$ ) a 82-91% yield of phenylsodium was obtained from benzene chloride and sodium in toluene solution. Phenyl sodium was transferred to a mixing flask containing 33 wt.%  $SiCl_4$  in toluene. After 1 hr of mixing, the reaction mixture was filtered in a  $N_2$  atmosphere and fractionally distilled at  $\leq 90$ , 90-170, 170-180, 180-220, 220-237, and 237-250°C. A 70-74% yield of triphenylchlorosilane was obtained in fractions at 220-250°C. Tetraphenylsilane (9-14%) and diphenyl-dichlorosilane (6-8%) were among the reaction products. The residue still contained 3.2% chlorine. A change of temperature from -30 to +20°C did not affect the yield.

Card 1/2

UDC: 546.281

L 39718-66

ACC NR: AP6007969

The highest yield was obtained when using the 3:1 ratio of  $C_6H_5Cl:SiCl_4$ . The authors thank S. S. Churanova for advice. Orig. art. has: 1 fig.

SUB CODE: 07/ SUBM DATE: none/ OTH REF: 009

Card

2/2/65

L 31889-66 EWT(m)/EWP(j)/T RM

ACC NR: AP6012529

(A)

SOURCE CODE: UR/0062/66/000/003/0475/0478

AUTHOR: Morozova, L. P.; Andrianov, K. A.; Morozov, N. G.; Golubtsov, S. A.

30  
B

ORG: none

TITLE: Formation of alkyl(aryl)chlorosilanes during direct reaction of alkyl(aryl)chlorides with silicon. Communication 5. Effect of secondary decomposition process of methyldichlorosilane on the synthesis of methylchlorosilanes

SOURCE: AN SSSR. Izvestiya. Seriya khimicheskaya, no. 3, 1966, 475-478

TOPIC TAGS: catalyst, methyldichlorosilane, silane, organic synthesis

ABSTRACT: It was found that in decomposition reactions of methyldichlorosilane the most active catalysts are those which possess high selectivity in the synthesis of methyldichlorosilane. When the activity of catalysts in the synthesis is increased so that the yield increases from 2.2 to 25.5 g of methyldichlorosilane per kg of mass per hour, the degree of decomposition of methyldichlorosilane under identical conditions also increases from 4.0 to 67.2% respectively. This is explained by the fact that both synthesis and decomposition of methyldichlorosilane occur on the same active centers. It was shown experimentally that the degree of decomposition of methyldichlorosilane in a stream of methyl chloride decreases by about 1 order of magnitude as compared with

UDC: 542.91+546.287+542.97

Card 1/2

L 31888-66 EWT(m)/EWP(j)/T RM

ACC NR: AP6012530

(A)

SOURCE CODE: UR/0062/66/000/003/0478/0482

AUTHOR: Popeleva, G. S.; Andrianov, K. A.; Golubtsov, S. A.

27  
B

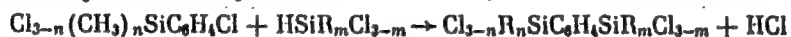
ORG: none

TITLE: Study of the reaction of methyl(chlorophenyl)chlorosilanes with hydrochlorosilanes

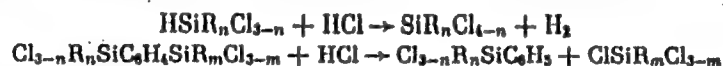
SOURCE: AN SSSR. Izvestiya. Seriya khimicheskaya, no. 3, 1966, 478-482

TOPIC TAGS: silane, organic synthesis, condensation reaction, substitution reaction

ABSTRACT: Using the previously described thermal condensation method [Authors Certificate No. 134699; Zh. obshch. khimii, 32, 557 (1962)] alkylchlorosilane hydrides were condensed with alkyl(chloroaryl)chlorosilanes by the following scheme:



where  $n=0, 1, 2, 3$ ;  $m=0, 1, 2$ . The condensation reaction is accompanied by a side reaction involving the reduction of chlorine in the aryl radical by the hydrogen of chlorosilane hydride. The products of substitution of hydrogen at the silicon by chlorine can be formed also by the decomposition reaction in hydrogen chloride medium as follows:



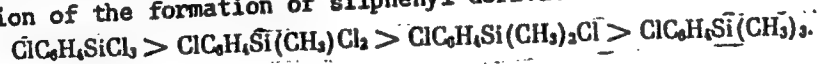
Card 1/2

UDC: 542.91+546.287

L 31888-66

ACC NR: AP6012530

Here in the formation of bis(methyldichlorosilyl)benzene decomposition and reduction reactions are approximately equivalent. It was shown that hydrogen can be substituted by chlorine, but also the organic radical connected to the silicon atom both in the starting materials as well as in the reaction products. The synthesized products are: bis(trichlorosilyl)benzene, bis(dimethylchlorosilyl)benzene, 1-methyldichlorosilyl-4-trichlorosilylbenzene, 1-dimethylchlorosilyl-4-trichlorosilylbenzene, 1-methyldichlorosilyl-4-dimethylchlorosilylbenzene, 1-trimethylsilyl-4-trichlorosilylbenzene and 1-trimethylsilyl-4-methyldichlorosilylbenzene. The best yield of silphenylene compounds was produced by chlorophenyltrichlorosilane ( $n=0$ ), but as  $n$  increases the yield of the principal product decreases. The investigated alkyl(chloroaryl)chlorosilanes are arranged in the following series in terms of their reactivity in the reaction of the formation of silphenyl derivatives:



Orig. art. has: 1 figure.

SUB CODE: 07/

SUBM DATE: 12Nov63/

ORIG REF: 006/

OTH REF: 008

LS  
Card 2/2

1 41315-65 EWI(m)/ENP(j) RM

ACC NR: AP6024019

SOURCE CODE: UR/0062/66/000/005/1009/1016

AUTHOR: Golubtsov, S. A.; Korobov, V. V. (Deceased); Popkov, K. K.; Trofimova, I. V.;  
Turetskaya, R. A.; Andrianov, K. A.; Belikova, Z. V.; Golosova, R. M.; Oggenblik, A. A.  
Aristova, V. G.

ORG: none

TITLE: Reactions of formation of alkyl(aryl)chlorosilanes in a direct interaction between alkyl (aryl) chlorides and silicon. Report No. 6. Role of cuprous chloride in the formation of dialkyldichlorosilanes

SOURCE: AN SSSR. Izv. Ser khim, no. 6, 1966, 1009-1016

TOPIC TAGS: silane, chloride, silicon compound, copper compound, *CHEMICAL REACTION*

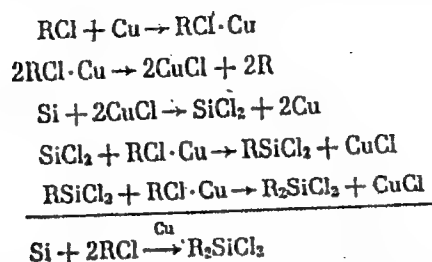
ABSTRACT: A mechanism is proposed for the formation of dimethyl(diethyl)dichlorosilane and methyl(ethyl)trichlorosilane during the reaction of methyl (ethyl) chloride with silicon on cuprous chloride. The proposed mechanism for the formation of dialkyldichlorosilanes is as follows:

Card 1/2

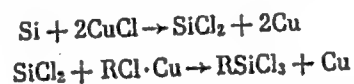
UDC: 546.287+542.91+541.124+543.422

L 415.5-65

ACC NR: AP6024019



The formation of alkyltrichlorosilane is represented as follows:



Experimental data obtained confirmed these mechanisms. Thermodynamic calculations of the initial stages of the reactions of methyl and ethyl chloride with silicon were performed. The formation of dichlorosilene is thermodynamically quite probable under the conditions of synthesis of alkylchlorosilanes. UV spectra of the products formed by the reaction of cuprous chloride with silicon showed a group of bands characteristic of the spectrum of  $\text{SiCl}_2$ . Orig. art. has: 2 figures and 5 tables.

SUB CODE: 07/ SUBM DATE: 12Feb64/ ORIG REF: 008/ OTH REF: 012

Card

2/2 *ML*



I. 43896-66 EWT(m)/EWP(j) RM  
ACC NR: AP6015624 (A)

SOURCE CODE: UR/0413/66/000/009/0024/0024

INVENTOR: Morozov, N. G.; Selik, G. I.; Andrianov, K. A.; Golubtsov, S. A.

ORG: none

TITLE: Method of obtaining methychlorosilanes. Class 12, No. 181105

SOURCE: Izobreteniya, promyshlennyye obraztsy, tovarnyye znaki, no. 9, 1966, 24

TOPIC TAGS: methylchlorosilane, methyl chloride, silane

ABSTRACT: An Author Certificate has been issued for a method of obtaining methylchlorosilanes by the interaction of methyl chloride with a silicon copper contact mass in the presence of an activator. To increase the content of trimethylchlorosilane in the mixture of terminal methylchlorosilanes, sodium aluminate chloride is used as the activator. [Translation] [NT]

SUB CODE: 11/ SUBM DATE: 24Feb65/  
07/

Card 1/1

UDC: 547.419.5.07

46180-66 EWT(m)/EWP(j) RM

ACC NR: AP6030559 (A,N) SOURCE CODE: UR/0413/66/000/016/0033/0033

INVENTOR: Ponomarev, V. V.; Shapatin, A. S.; Golubtsov, S. A.

ORG: none

TITLE: Preparative method for silicon-containing organophosphorus  
compounds. Class 12, No. 184856 ✓

SOURCE: Izobreteniya, promyshlennyye obraztsy, tovarnyye znaki, no. 16,  
1966, 33

TOPIC TAGS: *ORGANIC* phosphorus compound, silicon, alkylaryl silicon  
derivative, phosphorus trichloride, *CHEMICAL REACTION*

ABSTRACT: An Author Certificate has been issued for a method for pre-  
paring silicon-containing organophosphorus compounds of the general  
formula  $Cl_n R_n Si(R'PCl_2)_{4n-m}$ , where  $n = 0-3$ ,  $m = 0-3$ ,  $R$  is a monovalent  
alkyl or arylalkyl group, and  $R'$  is a bivalent alkylaryl group. The  
method involves the reaction of alkylaryl silicon derivatives of the  
 $R_n Cl_{3-n} Si(CH_2)_n C_6H_5$  type with  $PCl_3$  in the presence of Friedel-Crafts  
reaction catalysts, e.g.  $AlCl_3$ . [B0]

SUB CODE: 07/ SUBM DATE: 18May65/

Card 1/1 mjs

UDC: 547.419.1'5.07

L 42358-66 E-T(m)/EWP(j) RM  
ACC NR: AP6030555 (AN) SOURCE CODE: UR/0413/66/000/016/0033/0033

INVENTOR: Tsvanger, T. A.; Rostunov, V. F.; Golovnya, B. A.; Turetskaya, R. A.; Golubtsov, S. A.; Layner, D. I.; Malysheva, L. A.; Komrakova, V. V.; Yezerets, M. A.; Maslyukov, A. I.; Nastasin, A. A.

ORG: none

TITLE: Method of obtaining phenylchlorosilane. <sup>1</sup> Class 12, No. 184855.  
[announced by State Scientific Research Institute of State Design and Planning  
Scientific Research for the Processing of Nonferrous Metals (Gosudarstvennyy  
nauchno-issledovatel'skiy institut "Giprotsetmetobrabotka")]

SOURCE: Izobreteniya, promyshlennyye obraztsy, tovarnyye znaki, no. 16, 1966, 33

TOPIC TAGS: phenylchlorosilene, chlorobenzene

ABSTRACT: An Author Certificate has been issued for obtaining phenylchlorosilanes by the reaction of chlorobenzene with the silicon-copper contact mass in the presence of an activator. To raise the yield of diphenyldichlorosilane and to

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UDC: 547.419.5.07

1. 47358-66

ACC NR: AP6030558

increase the efficiency of the process, zinc oxide, in amounts up to 4%, is used as the activator. [Translation] [NT]

SUB CODE: 11/ SUBM DATE: 01Dec64/

Card 2/2 mt